EXPRESS MAIL NO.: EV343590577US
Attorney Docket No.: 108298765US
Client Ref. No.: 03-1044

AMPOULES FOR PRODUCING A REACTION GAS AND SYSTEMS FOR DEPOSITING MATERIALS ONTO MICROFEATURE WORKPIECES IN REACTION CHAMBERS

TECHNICAL FIELD

[0001] The present invention is related to ampoules for producing a reaction gas and systems for depositing materials onto microfeature workpieces in reaction chambers.

BACKGROUND

[0002] Thin film deposition techniques are widely used in the manufacturing of microfeatures to form a coating on a workpiece that closely conforms to the surface topography. The size of the individual components in the workpiece is constantly decreasing, and the number of layers in the workpiece is increasing. As a result, both the density of components and the aspect ratios of depressions (i.e., the ratio of the depth to the size of the opening) are increasing. Thin film deposition techniques accordingly strive to produce highly uniform conformal layers that cover the sidewalls, bottoms, and corners in deep depressions that have very small openings.

[0003] One widely used thin film deposition technique is Chemical Vapor Deposition (CVD). In a CVD system, one or more precursors that are capable of reacting to form a solid thin film are mixed while in a gaseous or vaporous state, and then the precursor mixture is presented to the surface of the workpiece. The surface of the workpiece catalyzes the reaction between the precursors to form a solid thin film at the workpiece surface. A common way to catalyze the reaction at

the surface of the workpiece is to heat the workpiece to a temperature that causes the reaction.

[0004] Although CVD techniques are useful in many applications, they also have several drawbacks. For example, if the precursors are not highly reactive, then a high workpiece temperature is needed to achieve a reasonable deposition rate. Such high temperatures are not typically desirable because heating the workpiece can be detrimental to the structures and other materials already formed on the workpiece. Implanted or doped materials, for example, can migrate within the silicon substrate at higher temperatures. On the other hand, if more reactive precursors are used so that the workpiece temperature can be lower, then reactions may occur prematurely in the gas phase before reaching the substrate. This is undesirable because the film quality and uniformity may suffer, and also because it limits the types of precursors that can be used.

[0005] Atomic Layer Deposition (ALD) is another thin film deposition technique. Figures 1A and 1B schematically illustrate the basic operation of ALD processes. Referring to Figure 1A, a layer of gas molecules A coats the surface of a workpiece W. The layer of A molecules is formed by exposing the workpiece W to a precursor gas containing A molecules and then purging the chamber with a purge gas to remove excess A molecules. This process can form a monolayer of A molecules on the surface of the workpiece W because the A molecules at the surface are held in place during the purge cycle by physical adsorption forces at moderate temperatures or chemisorption forces at higher temperatures. Referring to Figure 1B, the layer of A molecules is then exposed to another precursor gas containing B molecules. The A molecules react with the B molecules to form an extremely thin layer of solid material on the workpiece W. The chamber is then purged again with a purge gas to remove excess B molecules.

[0006] Figure 2 illustrates the stages of one cycle for forming a thin solid layer using ALD techniques. A typical cycle includes (a) exposing the workpiece to the first precursor A, (b) purging excess A molecules, (c) exposing the workpiece to the second precursor B, and then (d) purging excess B molecules. In actual

processing, several cycles are repeated to build a thin film on a workpiece having the desired thickness. For example, each cycle may form a layer having a thickness of approximately 0.5-1.0Å, and thus several cycles are required to form a solid layer having a thickness of approximately 60Å.

[0007] Figure 3 schematically illustrates an ALD system 1 including a single-wafer reaction chamber 10, a carrier gas supply 30, and an ampoule 60 in fluid communication with the reaction chamber 10 and the carrier gas supply 30. The reaction chamber 10 includes a heater 16 that supports the workpiece W and a gas dispenser 12 that dispenses gases into the reaction chamber 10. The gas dispenser 12 has a plenum 13 in fluid communication with the ampoule 60 and a distributor plate 14 with a plurality of holes 15. In operation, a carrier gas flows from the carrier gas supply 30 into the ampoule 60 and mixes with a precursor 70 to form a reaction gas. The reaction gas flows from the ampoule 60 to the gas dispenser 12 for deposition onto the workpiece W. The heater 16 heats the workpiece W to a desired temperature, and a vacuum 18 maintains a negative pressure in the reaction chamber 10 to draw the reaction gas from the gas dispenser 12 across the workpiece W and then through an outlet of the reaction chamber 10.

[0008] One drawback of ALD processing is that it has a relatively low throughput compared to CVD techniques. For example, each A-purge-B-purge cycle can take several seconds. This results in a total process time of several minutes to form a single thin layer of only 60Å. In contrast to ALD processing, CVD techniques require only about one minute to form a 60Å thick layer. The low throughput limits the utility of the ALD technology in its current state because ALD may create a bottleneck in the overall manufacturing process.

[0009] Another drawback of both ALD and CVD processing is that the precursors must be delivered in a gaseous state. Many potentially useful precursors, including, halides, THDs and DMHDs, are relatively low vapor pressure liquids or solids. It can be difficult to volatilize such precursors at a sufficient rate for a

commercially acceptable production throughput. Accordingly, a need exists to improve the process of vaporizing low volatility precursors.

BRIEF DESCRIPTION OF THE DRAWINGS

[0010] Figures 1A and 1B are schematic cross-sectional views of stages in ALD processing in accordance with the prior art.

[0011] Figure 2 is a graph illustrating a cycle for forming a layer using ALD techniques in accordance with the prior art.

[0012] Figure 3 is a schematic representation of a system including a reaction chamber for depositing materials onto a microfeature workpiece in accordance with the prior art.

[0013] Figure 4 is a schematic representation of a system for depositing materials onto a microfeature workpiece W in accordance with one embodiment of the invention.

[0014] Figure 5 is a schematic representation of an ampoule for use in processing microfeature workpieces in accordance with another embodiment of the invention.

[0015] Figure 6 is a schematic representation of an ampoule for use in processing microfeature workpieces in accordance with another embodiment of the invention.

[0016] Figure 7 is a schematic representation of an ampoule for use in processing microfeature workpieces in accordance with another embodiment of the invention.

[0017] Figure 8A is a schematic representation of an ampoule for use in processing microfeature workpieces in accordance with another embodiment of the invention.

[0018] Figure 8B is a top plan view of one of the trays in the precursor exposure assembly of Figure 8A.

[0019] Figure 9 is a top plan view of a precursor exposure assembly for use in an ampoule in accordance with another embodiment of the invention.

DETAILED DESCRIPTION

A. Overview

[0020] The following disclosure describes several embodiments of ampoules for producing a reaction gas and systems for depositing materials onto workpieces in reaction chambers. Many specific details of the invention are described below with reference to single-wafer reaction chambers for depositing materials onto microfeature workpieces, but several embodiments can be used in batch systems for processing a plurality of workpieces simultaneously. The term "microfeature workpiece" is used throughout to include substrates upon which and/or in which microelectronic devices, micromechanical devices, data storage elements, read/write components, and other features are fabricated. For example, microfeature workpieces can be semiconductor wafers such as silicon or gallium arsenide wafers, glass substrates, insulative substrates, and many other types of materials. Furthermore, the term "gas" is used throughout to include any form of matter that has no fixed shape and will conform in volume to the space available, which specifically includes vapors (i.e., a gas having a temperature less than the critical temperature so that it may be liquefied or solidified by compression at a constant temperature). Several embodiments in accordance with the invention are set forth in Figures 4-9 and the following text to provide a thorough understanding of particular embodiments of the invention. A person skilled in the art will understand, however, that the invention may have additional embodiments, or that the invention may be practiced without several of the details of the embodiments shown in Figures 4-9.

[0021] Several aspects of the invention are directed to ampoules for producing a reaction gas for processing microfeature workpieces in a reaction chamber. In one embodiment, an ampoule includes a vessel having an interior volume configured to receive a precursor with a headspace above the precursor. The ampoule further includes a carrier gas inlet for flowing carrier gas into the vessel, a conduit having an opening in the precursor and an outlet in the headspace, and

a flow driver for flowing the precursor through the conduit and into the headspace to increase the surface area of the precursor exposed to the carrier gas. The flow driver can include a pump or a carrier gas line configured to flow carrier gas into the conduit. The carrier gas entrains molecules of the precursor as the carrier gas flows into and through the conduit.

[0022] In another embodiment, an ampoule includes a vessel having an interior volume configured to receive a precursor with a headspace above the precursor. The ampoule further includes a conduit for conveying a flow of the precursor to the headspace, a carrier gas inlet for flowing carrier gas into the vessel, and a precursor exposure assembly at least partially within the headspace. The precursor exposure assembly is positioned so that at least some of the nonvaporized precursor flows from the conduit onto the assembly to increase the surface area of the precursor exposed to the carrier gas. The precursor exposure assembly can include a plurality of channels, a conical surface, and/or a plurality of trays arranged in a stack to hold discrete volumes of precursor.

[0023] Another aspect of the invention is directed to methods for processing microfeature workpieces in a reaction chamber. In one embodiment, a method includes delivering carrier gas to a vessel having a precursor and a headspace above the precursor, flowing the precursor through a conduit into the headspace to increase the surface area of the precursor exposed to the carrier gas, and removing from the headspace a reaction gas comprised of vaporized precursor. Flowing the precursor through the conduit can include entraining molecules of the precursor in a carrier gas that passes through the conduit. Alternatively, flowing the precursor through the conduit can include pumping the precursor through the conduit with a pump. The method can further include passing the reaction gas from the headspace to the reaction chamber and depositing a reaction product on a surface of the microfeature workpiece. The reaction product can be derived, at least in part, from the vaporized precursor.

B. Embodiments of Systems for Depositing Materials Onto Microfeature Workpieces

[0024] Figure 4 is a schematic representation of a system 100 for depositing materials onto a microfeature workpiece W in accordance with one embodiment of the invention. The illustrated system 100 includes a gas phase reaction chamber 110 for receiving the workpiece W, an ampoule 160 for carrying a precursor 170, and a carrier gas supply 130 for providing carrier gas to the ampoule 160 to facilitate transport of the precursor 170 to the reaction chamber 110. The carrier gas mixes with precursor 170 in the ampoule 160 to form a reaction gas. The reaction gas is delivered to the reaction chamber 110 and deposits a layer of material onto the surface of the workpiece W.

[0025] The illustrated system 100 further includes a carrier gas supply line 132 to convey the flow of carrier gas to the ampoule 160, and a reaction gas delivery line 140 to convey the flow of reaction gas from the ampoule 160 to the reaction chamber 110. Gas flow through the supply line 132 and the delivery line 140 can be regulated by one or more valves. For example, the gas flow can be regulated by an ampoule inlet valve 134, an ampoule outlet valve 142, a delivery line valve 144 (shown in hidden lines), and a chamber inlet valve 146 (shown in hidden lines). Optionally, a bypass line 136 (shown in hidden lines) with a valve 138 (shown in hidden lines) may deliver carrier gas directly from the supply line 132 to the delivery line 140 to control the concentration of the vaporized precursor in the reaction gas.

[0026] In some circumstances, more than one precursor may be necessary to deposit the desired reaction product on the workpiece W. A second precursor and a purge gas, for example, may be delivered from a second gas supply 150 (shown schematically and in hidden lines) and a third gas supply 155 (shown schematically and in hidden lines), respectively. The second gas supply 150 can be coupled to the reaction chamber 110 via a delivery line 152 (shown in hidden lines) having a valve 154 (shown in hidden lines) to control the flow of the second precursor, and the third gas supply 155 can be coupled to the reaction chamber

110 via a delivery line 156 (shown in hidden lines) having a valve 158 (shown in hidden lines) to control the flow of the purge gas. The first and second precursors can be the gas and/or vapor phase constituents that react to form the thin, solid layer on the workpiece W. The purge gas can be a suitable type of gas that is compatible with the reaction chamber 110 and the workpiece W. In other embodiments, the system 100 can include a different number of gas sources for applications that require additional precursors or purge gases.

[0027] The illustrated reaction chamber 110 includes a gas dispenser 112 to flow the gas(es) onto the workpiece W and a workpiece support 114 to hold the workpiece W. The workpiece support 114 can be heated to bring the workpiece W to a desired temperature for catalyzing the reaction between the first and second precursors at the surface of the workpiece W. For example, the workpiece support 114 can be a plate with a heating element. The workpiece support 114, however, may not be heated in other applications. A vacuum 116 (shown schematically) maintains negative pressure in the reaction chamber 110 to draw the gas(es) from the gas dispenser 112 across the workpiece W and then through an outlet of the reaction chamber 110.

[0028] The illustrated ampoule 160 includes a vessel 162 having an interior volume configured to receive the precursor 170 with a headspace 178 above the precursor 170. The vessel 162 should be made of a material that is relatively inert with respect to the precursor 170 such that contact between the precursor 170 and the interior surface of the vessel 162 does not unduly degrade the vessel 162 or contaminate the precursor 170. Moreover, the material of the vessel 162 should also be selected to withstand the rigors of use, which may include elevated processing temperatures, corrosive fluids, and/or friction with an abrasive particulate precursor. Suitable materials for forming the vessel 162 can include ceramics, glass, and metals such as stainless steel.

[0029] The illustrated ampoule 160 further includes a gas conduit 164 in fluid communication with the supply line 132 to convey a flow of carrier gas within the vessel 162. The gas conduit 164 has an opening 166 in the precursor 170 and an

outlet 168 in the headspace 178. The opening 166 is sized and positioned so that precursor 170 flows into the gas conduit 164 and becomes entrained in the carrier gas as the carrier gas flows through the conduit 164. Because the mixture of carrier gas and entrained precursor 172 is less dense than the liquid precursor 170, the entrained precursor 172 flows up the gas conduit 164 and through the outlet 168. As such, the portion of the gas conduit 164 between the opening 166 and the outlet 168 defines a lift tube 167 to convey a flow of entrained precursor 172 to the headspace 178. The lift tube 167 can have a hollow circular, rectangular, triangular, or other suitable cross-sectional configuration to convey the flow of entrained precursor 172.

[0030] In the lift tube 167, some of the entrained precursor 172 vaporizes as the precursor 172 is exposed to the carrier gas. The vaporized precursor is subsequently removed from the headspace 178 via the gas delivery line 140. The nonvaporized precursor 170 flows from the outlet 168 and back toward the precursor 170 at the base of the vessel 162. Additional amounts of the precursor 170 vaporize in the headspace 178 because the nonvaporized precursor 170 flowing between the outlet 168 and the liquid precursor 170 at the base of the vessel 162 is exposed to the carrier gas. As such, the precursor 170 is exposed to the carrier gas in the lift tube 167, at the surface of the liquid precursor 170, and in the external flow from the outlet 168 of the lift tube 167.

[0031] One feature of the ampoule 160 illustrated in Figure 4 is that the lift tube 167 increases the surface area of the precursor exposed to the carrier gas. Because the precursor is exposed to the carrier gas as the precursor flows through the lift tube 167 and from the outlet 168 toward the base of the vessel 162, the surface area of the precursor exposed to the carrier gas is greater than the transverse cross-sectional area of the vessel 162. An advantage of this feature is that the vaporization rate of the precursor in the vessel 162 is increased because the vaporization rate is generally proportional to the exposed surface area of the precursor. As such, certain low volatility precursors that do not vaporize in prior art ampoules at a sufficient rate for a commercially acceptable

production throughput may vaporize at commercially acceptable rates in the ampoule 160 illustrated in Figure 4.

[0032] Another feature of the ampoule 160 illustrated in Figure 4 is that the vaporization rate of the precursor is increased without increasing the flow rate of the carrier gas. An advantage of this feature is that the vaporization rate of the precursor is increased without reducing the concentration of precursor in the carrier gas.

C. Additional Embodiments of Ampoules for Use in Deposition Systems

[0033] Figure 5 is a schematic representation of an ampoule 260 for use in processing microfeature workpieces in accordance with another embodiment of the invention. The illustrated ampoule 260 is generally similar to the ampoule 160 described above with reference to Figure 4. The illustrated ampoule 260, however, includes a gas conduit 264 and a discrete lift tube 267 spaced apart from the gas conduit 264. The gas conduit 264 includes an outlet 265, and the lift tube 267 includes an opening 266 in the precursor 170 and an outlet 268 in the headspace 178. The lift tube 267 may also include a tapered portion 269 at the opening 266 so that the cross-sectional area of the opening 266 is greater than the cross-sectional area of the outlet 268.

[0034] The opening 266 of the lift tube 267 and the outlet 265 of the gas conduit 264 are positioned relative to each other so that carrier gas 263 flows from the outlet 265 into the lift tube 267. The carrier gas 263 entrains precursor 172 as the gas 263 flows through the lift tube 267, and some of the entrained precursor 172 vaporizes in the lift tube 267. Some of the nonvaporized precursor 170 may also vaporize as the precursor 170 flows from the outlet 268 of the lift tube 267 toward the liquid precursor 170 at the base of the vessel 162. As such, the illustrated lift tube 267 increases the surface area of the precursor exposed to carrier gas so that the ampoule 260 advantageously increases the vaporization rate of the precursor.

[0035] Figure 6 is a schematic representation of an ampoule 360 for use in processing microfeature workpieces in accordance with another embodiment of the invention. The illustrated ampoule 360 is generally similar to the ampoule 260 described above with reference to Figure 5. For example, the ampoule 360 includes a lift tube 367 with an opening 366 in the precursor 170 and an outlet 368 in the headspace 178. The illustrated ampoule 360, however, does not entrain precursor in a flow of carrier gas passing through the lift tube. Rather, the ampoule 360 includes a pump 369 (shown schematically) to flow the precursor 170 through the lift tube 367 and into the headspace 178. The pump 369 can be submerged in the precursor 170, positioned in the headspace 178, or located at another suitable position to flow precursor 170 through the lift tube 367. In the headspace 178, the precursor 170 is exposed to carrier gas, which is delivered to the vessel 162 via a carrier gas inlet 361. Exposure to the carrier gas causes some of the precursor 170 to vaporize. The nonvaporized precursor 170 flows back toward the liquid precursor 170 at the base of the vessel 162 for recirculation through the lift tube 367. As such, the illustrated ampoule 360 increases the surface area of the precursor 170 exposed to the carrier gas and, consequently, the vaporization rate of the precursor 170.

[0036] Figure 7 is a schematic representation of an ampoule 460 for use in processing microfeature workpieces in accordance with another embodiment of the invention. The illustrated ampoule 460 is generally similar to the ampoule 160 described above with reference to Figure 4. The illustrated ampoule 460, however, further includes a precursor exposure assembly 480 for increasing the surface area of the precursor 170 exposed to the carrier gas. The illustrated precursor exposure assembly 480 includes a conical member 482 having a surface 484 positioned proximate to the outlet 168 of the gas conduit 164 so that the nonvaporized precursor 170 falls onto the surface 484 after exiting the outlet 168. The slope of the conical member 482 conveys the flow of nonvaporized precursor 170 across the surface 484 in a direction S_1. While the nonvaporized precursor 170 flows across the surface 484, the precursor 170 is exposed to the

carrier gas in the headspace 178 and, consequently, some of the precursor 170 vaporizes. One advantage of the ampoule 460 illustrated in Figure 7 is that the precursor exposure assembly 480 increases the vaporization rate of the precursor 170 by increasing the exposure of the precursor 170 to the carrier gas.

[0037] Figure 8A is a schematic representation of an ampoule 560 for use in processing microfeature workpieces in accordance with another embodiment of the invention. The illustrated ampoule 560 is generally similar to the ampoule 160 described above with reference to Figure 4. The illustrated ampoule 560, however, includes a precursor exposure assembly 580 for increasing the surface area of the precursor 170 exposed to the carrier gas. The illustrated precursor exposure assembly 580 includes a plurality of trays 582 arranged in a stack and positioned proximate to the outlet 168 of the gas conduit 164. The trays 582 hold discrete volumes of precursor 170 to increase the surface area of the precursor 170 exposed to the carrier gas. In the illustrated embodiment, the nonvaporized precursor 170 flows from the outlet 168 of the gas conduit 164 and into a top tray 582a. As described in detail below, the individual trays 582 are configured so that cascading flows 176 of precursor 170 pass downward from one tray 582 to an adjacent tray 582. In other embodiments, the gas conduit 164 can include a plurality of holes to flow nonvaporized precursor 170 directly into the individual trays 582 in lieu of or in addition to the flow from the outlet 168 to the top tray 582a.

[0038] Figure 8B is a top plan view of one of the trays 582 of the precursor exposure assembly 580 of Figure 8A without the precursor 170. Referring to both Figures 8A and 8B, the illustrated trays 582 include a support surface 583, a hole 584 in the support surface 583, and an outer wall 586 projecting from the support surface 583. The hole 584 is sized to receive the gas conduit 164 (Figure 8A) so that the trays 582 can be arranged around the conduit 164. The outer wall 586 and the support surface 583 define an interior region configured to carry the nonvaporized precursor 170. The trays 582 can also include a plurality of notches 588 in the outer wall 586 through which the nonvaporized precursor 170

flows to an adjacent tray 582. More specifically, the outer wall 586 has a height H_1 (Figure 8A) and is positioned at a radius R_1 (Figure 8B) on the trays 582. The notches 588 have a height H_2 (Figure 8A) and are positioned at a radius R_2 (Figure 8B) less than the radius R_1. The trays 582 can be arranged with the notches 588 on adjacent trays 582 offset from each other so that the nonvaporized precursor 170 can flow downwardly into the adjacent tray 582. In additional embodiments, the precursor exposure assembly 580 can have other configurations. For example, the trays 582 may not include notches 588, and/or the individual trays may have different diameters. Moreover, the gas conduit 164 can be positioned to flow nonvaporized precursor 170 into the trays 582 without extending through the center of the tray stack.

[0039] One feature of the ampoule 560 illustrated in Figures 8A and 8B is that the trays 582 of the precursor exposure assembly 580 carry discrete volumes of precursor 170. An advantage of this feature is that the vaporization rate of the precursor 170 is increased due to the large surface area of the precursor 170 exposed to the carrier gas. Moreover, the trays 582 provide a relatively constant surface area that helps stabilize the vaporization rate of the precursor 170 and lends greater control to the concentration of the vaporized precursor 170 in the reaction gas extracted from the vessel 162.

[0040] Figure 9 is a top plan view of a precursor exposure assembly 680 for use in an ampoule in accordance with another embodiment of the invention. The illustrated precursor exposure assembly 680 includes a central member 681 and a plurality of channels 682 projecting radially outward from the central member 681. The central member 681 includes a hole 684 sized to receive a gas conduit so that the channels 682 can be positioned around the outlet of the gas conduit. The channels 682 can project radially outward and generally normal to the gas conduit, or alternatively, the channels 682 can project radially outward and downward toward the precursor at the base of the vessel. In either case, the channels 682 are configured to receive some of the nonvaporized precursor as it flows from the gas conduit to increase the surface area of the nonvaporized

precursor exposed to the carrier gas. The channels 682 can include a support surface 683 and sidewalls 686 projecting from the support surface 683. The sidewalls 686 ensure that the nonvaporized precursor flows across the support surface 683 in a direction S_2 so that the surface area of the precursor exposed to the carrier gas is predictable and consistent over time. In additional embodiments, the channels 682 may not have sidewalls 686 or may have other configurations.

[0041] From the foregoing, it will be appreciated that specific embodiments of the invention have been described herein for purposes of illustration, but that various modifications may be made without deviating from the spirit and scope of the invention. For example, any of the precursor exposure assemblies illustrated in Figures 7-9 can be used with any of the ampoules illustrated in Figures 4-6. Accordingly, the invention is not limited except as by the appended claims.